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Note

In-tandem methodology and recommendations for measuring soil hydraulic properties from saturation to dryness in laboratory conditions**

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A b s t r a c t. Laboratory determination of soil water retentions and hydraulic conductivity curves requires different techniques, depending on the number of data points needed. An important consideration in technique selection is soil sampling, particularly the distinction between disturbed and undisturbed samples, as laboratory methods can differ significantly for each kind of soil sample. This work addressed the increasing interest within soil physics in using methodologies that cover a wide range of water content and suction to determine soil hydraulic properties accurately. Achieving soil samples representative of physical processes is challenging due to the time required for measurements and the combination of techniques necessary to characterize soil water retention and hydraulic conductivity curves. A detailed workflow in-tandem for soil hydraulic curve measurements is presented, comprising more than 4 500 measurements to describe both curves from saturation to dryness. To enhance reproducibility and reduce uncertainty in laboratory measurement, this work offers ten best practice recommendations focused on sample preparation, instrument handling, and data management. This work aims to contribute to improved methodologies in soil physics laboratories and promote standardized practices. This work also contributes to advancing the reliability and accuracy of soil hydraulic properties assessment, thereby supporting improved research and application in soil science.

K e y w o r d s: laboratory techniques, soil hydraulic conductivity, soil hydrology, soil physics, soil-water retention curves

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1. INTRODUCTION

Hydrological models based on soil physical properties depend on soil hydraulic measurements for reliable estimates. Soil water retention (SWR) and hydraulic conductivity (HC) curves are needed for many irrigation, drainage, water movement, and solute transport models in soils (Shwetha and Varija, 2015). The soil's saturated hydraulic conductivity (K_s) is commonly measured using a constant or falling head method on cylindrical core samples (Deb, 2012). The SWR (wet range) and HC curves (unsaturated soil) are often measured simultaneously by the simplified evaporation method (SEM) (Schelle *et al.*, 2013). The SEM records pressure heads, water contents, and evaporation fluxes, estimating water retention and unsaturated hydraulic conductivity (Lipovetsky *et al.*, 2020). Laboratory evaporation experiments, such as SEM, are particularly popular because they simultaneously provide data for SWR and HC curves (Inforsato *et al.*, 2023). The dry range of the SWR curve can be obtained by the chilled mirror dewpoint technique (Schelle *et al.*, 2013). Thus, no single laboratory device can measure over the entire range of SWR and HC curves (Haghverdi *et al.*, 2018), so a combination of methods is needed.

Although there are studies that show the use of SEM, dewpoint method, and K_s measurements in the same research (Haghverdi *et al.*, 2018; Lipovetsky *et al.*, 2020; Hohenbrink *et al.*, 2023), the lack of standardization in the experimental conditions related to soil hydraulic properties contributes to a lack of reproducibility between different laboratories employing different methods or the same methods with different procedures (Guillaume *et al.*, 2023). This work focuses on methodologically detailing how to obtain soil hydraulic properties (SWR and HC curves) from saturation to dryness, and it is focused on optimizing the use of limited laboratory resources and reducing sample variability. Therefore, a laboratory setup and workflow for obtaining both SWR and HC curves from the same soil sample and ten detailed recommendations for the steps are described as follows.

2. METHODOLOGY

The proposed methodology is based on the collected data from 46 different sites in Chile. Almost all sites were samples in duplicate, but six sites were sampled in triplicate. This means 98 soil samples and more than 4 500 measurements have been taken using laboratory equipment since 2017. To illustrate the methodology to ensure reproducibility for obtaining SWR and HC curves, this work included sixteen soils covering six of twelve USDA textural classes. The soil samples were measured in duplicate for testing in-tandem methodology. Figure 1 shows the steps for the methodology, which are explained below.

2.1. Proper laboratory conditions

Before starting measurements of soil hydraulic properties, controlled laboratory conditions (constant temperature and humidity) are needed. The SEM assumes linearity of the evaporation rate with time (Peters *et al.*, 2015); hence, a constant evaporation rate from the soil sample must be ensured. The evaporation rate is given by the atmospheric demand of the laboratory air, and keeping a constant vapor pressure deficit helps to ensure the constant evaporation rate (Peters and Durner, 2008).

2.2. Soil core sampling

Undisturbed soils from the field are preferred over repacked soils to represent the soil structure better. To ensure soil samples are undisturbed and as similar as possible to field conditions, field soil cores are taken from the field using a 250 cm^3 sampling ring (5 cm in height and 8 cm in diameter), using a rubber hammer, and then wrapping the soil sample in the cylinder in cheesecloth. The wrapped cylinder is placed in plastic or metal containers for safe transportation. Then, soil samples are refrigerated (4°C) until analysis. Larger metallic rings are preferred over smaller ones. The larger diameter of the ring reduces the edge effect and better represents the soil volume.

2.3. Soil core storage

Soil cores from the field have to be stored between collection and soil hydraulic measurements. A fresh analysis is ideal, but that can be difficult to achieve due to equipment usage time and the number of samples being analyzed in parallel. A common way to store soil cores for soil hydraulic measurements is to wrap them in aluminum foil or cheesecloth and then, the wrapped soil cores are stored in individuals' plastic containers at 4°C until analysis. Storing and refrigerating the samples at 4°C can minimizes the occurrence of mold in the soil sample surface. If the must be stored longer, a small amount of ethanol can be sprayed on the soil sample surface (Acevedo *et al.*, 2023).

2.4. Soil characterization

Before soil hydraulic properties are measured, the following soil properties should be measured: Organic matter (OM) content, particle-size distribution (PSD), and electrical conductivity (EC). In this work, the OM content, EC,

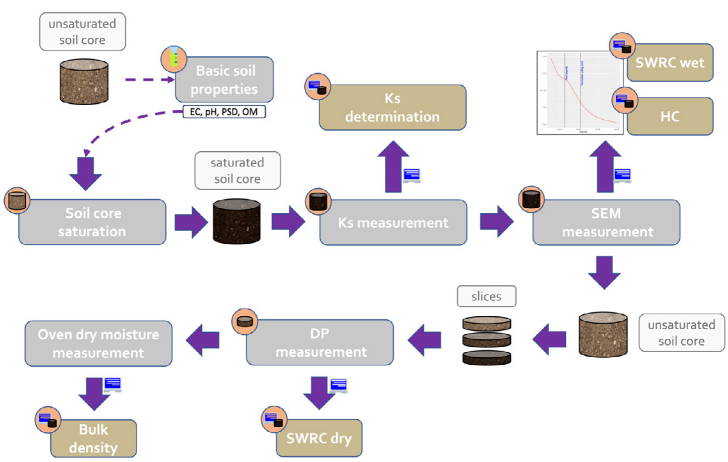

Fig. 1. Steps of the proposed in-tandem methodology. All the steps in the diagram use the sample soil core sample. The process starts with the unsaturated soil sample, obtaining the basic soil properties, and then soil core saturation. Grey rectangles indicate the processes, and the brown rectangles indicate the data obtained from the process.

and soil texture were measured using the Walkley-Black, the 1:5 soil:water solution, and hydrometer methods, respectively, following the guidelines for Chilean soils (Acevedo *et al.*, 2021). However, it is important to point out that each country has its own soil guidelines for measuring OM, EC and soil texture.

The OM content, PSD, EC, and soil salinity are some properties that control soil water retention and K_s, so knowing them in advance allows for estimating the measuring time and variables that may affect the measurement.

2.5. Soil core repacking

Among the storage protocols recommended for soil, air-drying and storage at room temperature are the most used for non-biological conservation (Turner and Romero, 2010). This sieved soil (< 2 mm) can be repacked and measured (Haghverdi *et al.*, 2018). If disturbed samples are used for measurements, to repack them, small discrete amounts of air-dried soil must be added into the sampling ring and then mechanically packed with a flat pestle (Lewis and Sjöstrom, 2010) until they reach a similar bulk density observed in the field. Scarifying the soil surface after compaction and before the addition of another lift can ensure hydraulic connectivity between the layers (Plummer *et al.*, 2004).

2.6. Soil core saturation

Before saturation, soil cores must be taken out from the refrigerator and covered with cheesecloth on the bottom. Saturation is done by immersing the soil sample cores in a plastic pan containing degassed $CaCl_2$ solution with the same soil EC. Degassing can be done by vacuuming or boiling water prior to preparing the solution. To saturate the soil sample, the water level has to be close to the rim of the soil ring (Araya *et al.*, 2022). A gloss on the upper surface indicates that the soil sample is fully saturated if the saturation process was done from the bottom upwards by capillarity (Shokrana and Ghane, 2020). During the saturation phase, some biofilms, sprouts, and fungi can appear on the upper surface of the soil cores due to biological activity facilitated by the saturation condition. Do not saturate the sample longer than necessary, and use solutions prepared with boiled water to avoid the growth of microorganisms. If needed, add a small amount of antibiotic or ethanol, making sure not to disturb the soil sample or change the EC.

2.7. Saturated core handling

Once the soil core is saturated, it can be measured on the KSat device to obtain K_s. Due to the importance of the saturation process in obtaining a correct measurement, the analysis must be done before handling the soil core to avoid desaturation while handling. In the KSat setup, the gap between the soil core and aluminum ring must be filled with a metal mesh or porous plate, depending on how coarse the sample is. The saturated cores should be set up so that the flow direction is downward in KSat and the evaporation upward in SEM (Peters *et al.*, 2015; Araya *et al.*, 2022). This means that for KSat measurement, the sharp end of the soil ring must be facing upwards.

2.8. From simplified evaporation to dewpoint method

The SEM and dewpoint methods can be combined using the same core sample (Kirsten *et al.*, 2019; Schelle *et al.*, 2013) to obtain an SWR curve covering the full moisture range. Two holes are drilled in the soil core for the SEM using a HYPROP device (Meter Group, Pullman, WA, USA). The holes are made using a mini auger to insert both tensiometers. The mini auger must be removed slowly and gently to not compact the soil contact area with the tensiometer. The measurements (water loss in the soil core vs. time) start with the tensiometer's suctions close to zero; the difference between them is less than 3 hPa. The measurements are recorded until the suction values in both tensiometers fall close to zero after 5 or 7 days, depending on the soil sample. In the SEM, the measurement suction ranges from 0 hPa to 10 000 hPa, and the final measurement can be estimated to be close to 100 000 hPa.

The evaporation method depends on the precision weighting of the evaporation loss because every point is calculated by dividing the mass of the soil water by the volume of the soil body. Periodic checking of balance accuracy and precision must be part of the quality control process for measuring SWR curves. The proposed setup recommends an enclosure around the SEM because measurements with a resolution of 0.001 gram can be affected by airflow. Also, balances are extremely sensitive to vibration or movement, so using an especially sturdy table and minimizing movement around the SEM measurements are recommended.

Once the SEM measurement is stopped, Kirsten *et al.* (2019) proposed slicing the soil core into slabs (three to five slabs) and applying the dewpoint method to the slabs' subsamples. The dewpoint is done using the WP4C DewPoint Potentiometer (Meter Group, Pullman, WA, USA). This method has the advantages of using the same sample and using drying instead of wetting (avoiding uncertainty due to hysteresis). The dewpoint method allows measurements over a suction of 100 000 hPa. The only disadvantage is that the subsamples must be handled carefully so as not to lose weight and correctly calculate the oven-dry mass. The soil core's water content and bulk density were measured after oven-drying at 105°C, obtaining the sample bulk density in the same process. The resulting soil weight can be added to the software.

2.9. Data quality check

After the measurements, data hygiene must be performed manually in the HYPROP-FIT software before exportation to .csv format. Before exporting the data, it should be checked if the initial water content was smaller than the porosity (Hohenbrink *et al.*, 2023). It is also needed to check if the temperature of the devices did not have any important variation during the measurement, and it is also needed to check if the dewpoint measurements are within its valid range (pF between 4.0 and 6.5).

2.10. How to manage the obtained data

Soil physical properties analysis must include information from the field and laboratory due to their high variability. Different programming languages, including R, MATLAB, and Python, can be used for data analysis of the soil hydraulic properties. Scripts can help perform reproducible research using the primary data files, which should come from an untouched data store (Broman and Woo, 2018; Acevedo *et al.*, 2023). In this work, soil data visualization was plotted using ggplot2 (Wickham, 2016) and the ggsoiltexture R packages (Acevedo, 2024). This allows reproducibility over time.

3. RESULTS, ANALYSIS, AND FURTHER APPLICATION

The covered soil texture classes to illustrate the in-tandem methodology were clay (n = 1), clay loam (n = 4), loam (n = 6), sandy clay loam (n = 2), sandy loam (n = 2), and silty clay (n = 1). The OM content in soil samples ranged from 0.72 to 10.6%, with a mean of 4.15%. Figure 2 shows the soil texture covered in this study (a), the SWR data points (b), and the HC data points obtained (c) with this methodology. Regarding water content, the sandy loam sample with the higher OM content exhibited higher volumetric water content in the wet part of the SWR curve. This observation aligns with the findings of Minasny and McBratney (2018), who reported that the relationship between OM and water retention is generally weak, with notable effects primarily near field capacity in sandy soils and minimal impact in finer soil textures. In soils with high OM content (>10%), K_s is strongly affected by the humification degree, with well-humified matter being much less permeable than fibrous matter (Balland *et al.*, 2008). Clay soils exhibit seasonal variations in pore geometry and are particularly susceptible to disturbances because flow processes are governed by only a few larger pores. Due to soil structural heterogeneity, clay samples require large samples and replicates (Baker and Bouma, 1976). Clayey sodic soils can require hundreds of pore volumes of leaching to achieve a stable measurement, and early measurements can be translated into a tenfold overestimation of K_s (Reading *et al.*, 2015).

It is important to note that soils with higher OM and higher clay content required a longer time for analysis due to the prolonged saturation process and the extended duration of the SEM. Soil samples that took the longest time to measure in the SEM were one sandy loam soil, 12.5 days, and OM = 10.6%, and one loam soil, 10.5 days, and OM = 7.6%. Surprisingly, the clay soil took six days on average, less than the sandy loam and loam soil mentioned before. The OM of the clay soil was 2.1%, less than the other soils. In general, soil with high OM content tended to take more time when measuring with the SEM, but finer soil, meaning soils with high clay or silt content, took more time in the saturation process.

Some researchers have been successful in optimizing SEM measurement conditions using chambers, air fans, and heat sources (Tran *et al.*, 2019). In this study, the laboratory conditions allowed us to keep a constant vapor pressure deficit. Specifically, the laboratory conditions were a temperature of 23±1°C, and relative humidity was around 23±1%. Decreasing the temperature increases the length of time needed for SEM measurement. Matric

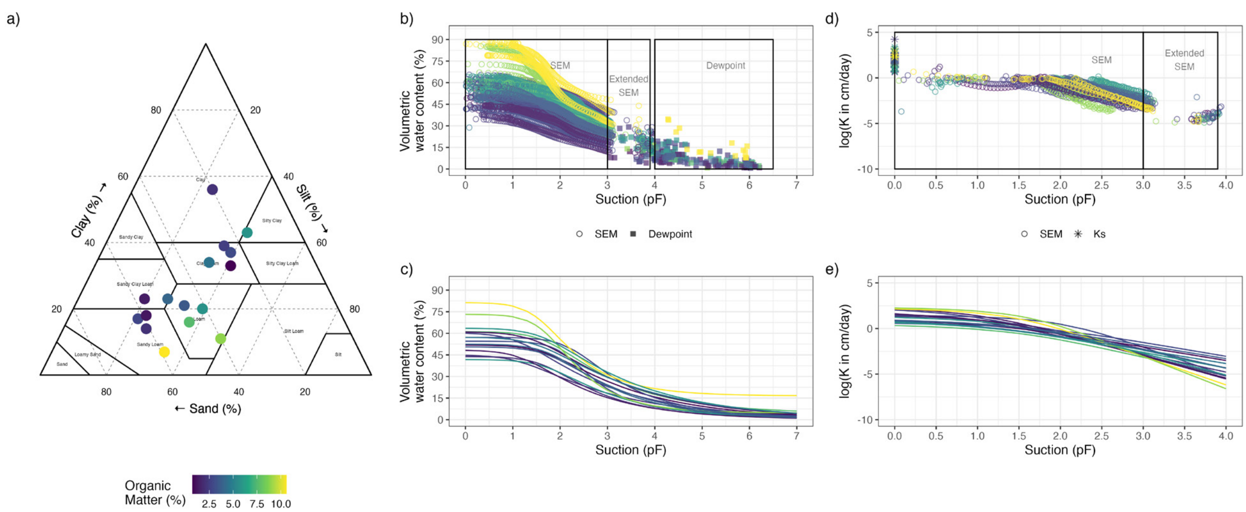

Fig. 2. Soil texture triangle a), SWR b) and c), HC d) and e) curves for 16 soils. The color (from blue to yellow) represents the sample OM content (mean = 4.15%, minimum = 0.72%, maximum = 10.6%). SEM ranges are from pF = 0 to pF < 4 (including extended SEM), and the dewpoint method ranges from pF > 4.2. Circles, squares, and asterisks represent the measurements b) and d), solid lines indicate curve fitting c) and e).

potential and soil water surface tension depend on temperature, making HC and K_s measurements sensitive to the temperature (Jiang *et al.*, 2021). It has been reported that the presence of biofilm can reduce the K_s by up to one order of magnitude (Volk *et al.*, 2016), so the organism's growth could cause misreading. Also, soil hydraulic conductivity tends to decrease significantly over time when the saturation solution contains microorganisms (Reading *et al.*, 2015). Some treatments suppress biological activity used in packed soil columns for K_s analysis, including phenol and NaClO, which are able to decrease pore-clogging by biological activity; however, they can alter cementation and soil structure. Thus, saturating the soil samples only for the time necessary, which can be estimated knowing the soil texture, is mandatory for proper sample handling. Additionally, if repacking soil samples is necessary, dry repacking is preferable as it is easiest to keep the same bulk density as in the field. Previous research has shown wet repacking produces a highly homogenous core, but some solutes may be lost during the filling process (Ikoyi and Schmalenberger, 2021). If ion composition is altered, then the measurement of hydraulic conductivity can be affected.

Although the SEM is typically concluded when both tensiometers reach zero, measurements can be stopped when only one tensiometer reaches zero. In this case, it is important to consider the soil sample's moisture before the analysis with the dewpoint method since this method measures the potential in the dry part of the curve (pF > 4.2). The dewpoint method assumes the osmotic potential is negligible compared to the matric potential, so the measured points can be added to the points obtained by SEM. The SEM provides around one hundred data points at lower suction values in contrast to 5 values by pressure plates method in a similar time frame of two weeks (Fields *et al.*, 2016), and it also allows, in parallel, the obtaining of the HC curve. The number of data points obtained from the SEM, the dewpoint methods, and the methods to obtain saturated hydraulic conductivity will allow a better fitting of functions later, depending on the user's needs. It is important to note that the accuracy of the methods depends on the laboratory setup and proper conditions in terms of temperature and humidity.

Obtaining the raw data for fitting a new function or obtaining parameters for available water in soils or contaminant movement in soil relies on data checking and management. In this work, more than 350 .csv and .xlsx files were generated. All the data were managed in R, allowing the creation of new data frames containing the information on the suction and the water retention (in %) and hydraulic conductivity curve (log K in cm d^{-1}) in the same date frame. The management of the data from the soil hydraulic properties' curves allowed the analysis with other soil properties, such as particle size distribution or OM content. This recommended management allows reproducibility over time since the data is stored in the original files, and the coding process creates a new data frame to process all the data obtained.

4. CONCLUSIONS

Soil physical properties are key descriptors in agriculture, environmental management, and civil engineering. Therefore, robust and reliable methodologies are needed. The methodology outlined in this work, informed by the processing of over 90 soil samples and a review of recent literature, offers detailed guidance for each stage from an operator's perspective. This work is valuable for researchers and laboratories equipped with described methodologies who wish to implement them sequentially, as well as for those evaluating which techniques to adopt for comprehensive soil physical studies using a single core sample.

The work recommended a standardized procedure from soil sampling to data management and different laboratory equipment according to the data needed. This work showed the successful obtainment of soil water retention and the hydraulic conductivity curves from the pF = 0 to pF > 5, combining different methods to obtain both curves, such as the falling and constant head method for the hydraulic conductivity curve, simplified evaporation method for both water retention and hydraulic conductivity curves and the dewpoint method for water retention curve.

The approach used in this work showed how soil hydraulic properties can be obtained using the same core soil sample, reducing field soil sampling efforts and preparation time in a laboratory while minimizing uncertainty associated with using different soil samples. A remarkable aspect is the potential independent use of each laboratory equipment depending on the user's needs. This guidance will assist soil scientists in selecting the most appropriate methodology and understanding the time requirements for processing each soil sample, alongside ten best practice recommendations for accurate laboratory analysis.

Declaration of competing interest. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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